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PATENT APPLICATION
TRANSMITTAL**

(Only for new nonprovisional applications under 37 C.F.R. § 1.53(b))

Attorney Docket No.	06061 USA
First Inventor or Application Identifier	Jeffrey Kramer
Title	Catalysts Which Stabilize Hydrofluorocarbon Blowing ...
Express Mail Label No.	TB827974873US

APPLICATION ELEMENTS
See MPEP chapter 600 concerning utility patent application contents.ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. ☒ * Fee Transmittal Form (e.g., PTO/SB/17)
(Submit an original and a duplicate for fee processing)
2. ☒ Specification [Total Pages 10]
(preferred arrangement set forth below)
- Descriptive title of the Invention
- Cross References to Related Applications
- Statement Regarding Fed sponsored R & D
- Reference to Microfiche Appendix
- Background of the Invention
- Brief Summary of the Invention
- Brief Description of the Drawings (if filed)
- Detailed Description
- Claim(s)
- Abstract of the Disclosure
3. ☐ Drawing(s) (35 U.S.C. 113) [Total Sheets ☐
4. Oath or Declaration [Total Pages ☐
 a. ☒ **Unsigned**
 b. ☐ Copy from a prior application (37 C.F.R. § 1.63(d))
 i. ☐ **DELETION OF INVENTOR(S)**
 Signed statement attached deleting
 inventor(s) named in the prior application,
 see 37 C.F.R. §§ 1.63(d)(2) and 1.33(b).

5. ☐ Microfiche Computer Program (Appendix)
6. Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
 a. ☐ Computer Readable Copy
 b. ☐ Paper Copy (identical to computer copy)
 c. ☐ Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

7. ☐ Assignment Papers (cover sheet & document(s))
8. ☐ 37 C.F.R. § 3.73(b) Statement ☐ Power of Attorney
(when there is an assignee)
9. ☐ English Translation Document (if applicable)
10. ☐ Information Disclosure Statement (IDS)/PTO-1449 ☐ Copies of IDS Citations
11. ☐ Preliminary Amendment
12. ☒ Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
13. ☐ * Small Entity Statement(s) ☐ Statement filed in prior application, Status still proper and desired (PTO/SB/09-12)
14. ☐ Certified Copy of Priority Document(s)
(if foreign priority is claimed)
15. ☐ Other: _____

* NOTE FOR ITEMS 1 & 13: IN ORDER TO BE ENTITLED TO PAY SMALL ENTITY FEES, A SMALL ENTITY STATEMENT IS REQUIRED (37 C.F.R. § 1.27), EXCEPT IF ONE FILED IN A PRIOR APPLICATION IS RELIED UPON (37 C.F.R. § 1.28).

16. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in a preliminary amendment:

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No: _____

Prior application information: Examiner _____ Group / Art Unit: _____

For CONTINUATION or DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 4b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

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Name

23543

Air Products and Chemicals, Inc.

Address

City

State

Zip Code

Country

Telephone

Fax

Name (Print/Type)

Michael Leach

Registration No. (Attorney/Agent)

27,349

Signature

Date

7 August 2000

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CATALYSTS WHICH STABILIZE HYDROFLUOROCARBON BLOWING AGENTS IN POLYURETHANE FOAM COMPOSITIONS

FIELD OF THE INVENTION

This invention relates to closed cell polyurethane foams expanded with hydrofluorocarbons in the presence of specific polyurethane catalysts which also serve to maintain the chemical integrity of the hydrofluorocarbon during exposure of the foams
5 to temperatures above ambient temperature.

BACKGROUND OF THE INVENTION

It is well known to those skilled in the art that polyurethane foams can be prepared by reacting and foaming a mixture of ingredients, consisting in general of an
10 organic polyisocyanate and an appropriate amount of polyol or mixture of polyols in the presence of a volatile liquid blowing agent, which is caused to vaporize by the heat liberated during the reaction of the polyisocyanate and polyol. It is also well known that this reaction and foaming process can be enhanced through use of amine and metal carboxylate catalysts as well as surfactants. The catalysts ensure adequate curing of
15 the foam while the surfactants regulate and control cell size.

In the class of foams known as low density, rigid polyurethane foam, the blowing agents of choice early on had been trichlorofluoromethane and dichlorodifluoromethane, known in the art as CFC-11 and CFC-12, respectively.

Due to the concern over the depletion of the ozone layer by chlorine atoms
20 introduced to the atmosphere from the release of fully halogenated chlorofluorocarbons (CFCs), alternative blowing agents have been sought. Hydrochlorofluorocarbons

(HCFCs) such as HCFC-141b have been allowed temporarily to displace the CFCs. The HCFCs will in turn be phased out in favor of more environmentally acceptable hydrofluorocarbons (HFCs).

However, organic substances which bear a hydrogen on one carbon and a
5 fluorine on an adjacent carbon will undergo elimination reactions under the influence of acids or bases to produce fluoroalkenes and hydrogen fluoride or products from the combination of the hydrogen fluoride with the base, known as salts.

Therefore, in view of the fact that some of the major and many of the minor components, e.g. polyols and catalysts, are of known basic character, dehydro-
10 fluorination of the HFCs might occur. Many of these fluoroalkenes possess unknown properties and it is therefore desirable to hold their formation to a minimum as a precautionary measure.

US 5,668,834 discloses catalysts which stabilize hydrohalocarbon blowing agents in polyurethane foam formulations.
15

SUMMARY OF THE INVENTION

The use of certain catalysts, in addition to catalyzing the gelling, or polymerization, reaction of polyisocyanates and polyols and the blowing reaction of polyisocyanate and water, result in a decreased amount of decomposition of
20 hydrofluorocarbon blowing agent to fluoroalkene during the reactions, and especially during use of the foam at elevated temperatures, such as temperatures of at least 54°C.

The present invention provides compositions comprising polyisocyanate, polyol, hydrofluorocarbon blowing agent, optionally water as an additional blowing agent, surfactant and at least one catalyst for polymerizing the polyisocyanate and polyol, i.e.,
25 the gelling reaction, and/or one catalyst for enhancing the blowing reaction of water with the polyisocyanate to generate carbon dioxide blowing agent.

Also provided is a method for making polyurethane foam comprising reacting a polyisocyanate with a polyol in the presence of a hydrofluorocarbon blowing agent, optionally water as an additional blowing agent, a surfactant and at least one catalyst for polymerizing the polyisocyanate and polyol, i.e., the gelling reaction, and/or one catalyst
5 for enhancing the blowing reaction of water with the polyisocyanate.

The gelling catalysts for use in the invention include triethylenediamine (TEDA); N-2-hydroxypropyl-triethylenediamine ammonium salt; N-cetyl-N,N-dimethylamine; N,N-diethyl-ethanamine; N,N-dimethylaminoethylmorpholine; bis(3-dimethylamino-propyl)-N,N-dimethylpropanediamine; N-cyclohexyl-N-methylcyclohexylamine; 1,3,5-
10 tris(3-(dimethylamino)propyl)hexahydro-s-triazine; bis(dimethylaminopropyl)methylamine; dibutyltin dilaurylmercaptide; dibutyltin diisooctylmaleate; dibutyltin bis(2-ethylhexyl-mercaptoacetate); stannous octoate and 1,2-dimethylimidazole.

Suitable blowing catalysts for use in the invention include bis-(dimethylamino-ethyl)ether; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine and bis(N,N-
15 dimethylaminoethyl)ether.

The use of these catalysts provides for a reduction in the decomposition of the HFC blowing agents to fluoroalkenes.

DETAILED DESCRIPTION OF THE INVENTION

20 The invention provides a method and composition for making HFC-blown polyurethane foam in which specific catalysts are used to reduce the decomposition of the HFC blowing agent. The method comprises reacting a polyisocyanate and a polyol in the presence of an HFC blowing agent, optionally water as an additional blowing agent, the specified catalyst(s) and surfactant cell stabilizer.

25 The gelling catalysts for use in the invention include triethylenediamine; N-2-hydroxypropyl-triethylenediamine ammonium salt; N-cetyl-N,N-dimethylamine;

N,N-diethyl-ethanamine; N,N-dimethylaminoethylmorpholine; bis(3-dimethylamino-propyl)-N,N-dimethylpropanediamine; N-cyclohexyl-N-methylcyclohexylamine; 1,3,5-tris(3-(dimethylamino)propyl)hexahydro-s-triazine; bis(dimethylaminopropyl)methylamine; dibutyltin dilaurylmercaptide; dibutyltin diisooctylmaleate; dibutyltin bis(2-ethylhexyl-mercaptoacetate); stannous octoate and 1,2-dimethylimidazole. The preferred gelling catalysts are triethylenediamine; N-2-hydroxypropyl-triethylenediamine ammonium salt; N-cetyl-N,N-dimethylamine; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine; N-cyclohexyl-N-methylcyclohexyl-amine; 1,3,5-tris(3-(dimethylamino)propyl)hexahydro-s-triazine; and bis(dimethylamino-propyl)methylamine.

10 Suitable blowing catalysts for use in the invention include bis-(dimethylamino-ethyl)ether; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine and bis(N,N-dimethylaminoethyl)ether.

In addition to the above amine gelling and blowing catalysts, also useful would be their acid blocked derivatives, i.e. the amine compounds existing as the salt with an organic acid, such as formic acid, acetic acid, 2 ethylhexanoic acid and the like.

15 The foregoing amine and tin catalysts are well known in the art and may be obtained commercially from urethane catalyst suppliers such as Air Products and Chemicals, Inc.

At least one catalyst is used in the present invention. Depending upon the catalyst selected, mixtures of two or more catalysts may be desirable.

The amount of the catalyst employed in the invention will vary depending upon the catalyst selected, the application, the type of foam being prepared, the identity of the polyol and other factors, but can readily be determined by anyone skilled in the art. The catalyst is used in an amount sufficient to catalyze the reaction of the polyisocyanate and the polyol and/or water, and also results in a decreased amount of decomposition of the hydrofluorocarbon blowing agent to fluoroalkene during the polymerization. Preferably,

the catalyst is present at an amount of at least 0.1 parts by weight per hundred parts of polyol (pphp). More preferably, the catalyst is present at an amount of 0.1 to 5 pphp.

In accordance with this invention, any of these catalysts or combinations of these catalysts may be used to prepare a variety of polyurethane foams by standard
5 techniques known in the art which may include the use of various standard additives such as surfactants, water, fire retardants, and others.

Typically used ratios of polyisocyanate to polyol and of a blowing agent to those components may be used in practicing the invention. The suitable polyisocyanates, polyols and surfactant cell stabilizers are those that can be found in any of the
10 polyurethane foam patent literature including US 5,688,834; 5,824,711; and 5,852,065 which are incorporated by reference.

The use of the catalyst will provide rigid foams with significantly reduced levels of fluoroalkenes formed during polymerization and especially during foam use at elevated temperatures compared to levels of fluoroalkenes found when using catalysts commonly
15 used in the rigid foam industry such as N,N-dimethylcyclohexylamine.

In practicing the invention, the molar ratio of polyisocyanate to polyol is about 1.1:1. As those skilled in the art know, the use of polyisocyanate to polyol in an amount of about 1.1:1 results in the formation of polyurethane foams.

Any hydrofluorocarbon blowing agent may be used in the present invention.
20 Such hydrofluorocarbon blowing agents include 1,1-difluoroethane; 1,2-difluoroethane; 1,1,1-trifluoroethane; 1,1,2-trifluoroethane; 1,1,1,2-tetrafluoroethane; 1,1,2,2-tetrafluoroethane; 1,1,1,2,2-pentafluoroethane; 1,1,1,3-tetrafluoropropane; 1,1,2,3,3-pentafluoropropane; and 1,1,1,3,3-pentafluoro-n-butane. The preferred HFC blowing agents are 1,1,1,3-tetrafluoropropane (HFC-245fa) and 1,1,1,2-tetrafluoroethane (HFC-
25 134a).

Mixtures of the HFC blowing agents with water and/or with hydrocarbon blowing agents such as pentane may also be used in the present invention. Also useful may be mixtures of the preferred hydrofluorocarbon blowing agents HFC-245fa and HFC-134a.

The term "fluoroalkene" is used to mean those organic compounds having at least one double bond and at least one fluorine atom. The fluoroalkenes which form depend on the hydrofluorocarbon blowing agent used. For example, the blowing agent 1,1,1,3,3-pentafluoropropane (HFC-245fa) would decompose to 1,3,3,3 tetrafluoropropene (HFC-1234ze).

10

EXAMPLE 1

This example shows the use of specified catalysts in the HFC-245fa blown rigid polyurethane foam formulation of Table 1.

Table 1

Component	Parts by Weight
Polyol ^a	100
Catalysts	2.6
Water	2.0
HFC-245fa ^b	34.2
Silicone Surfactant	3.0
Polymeric MDI	NCO Index = 115

15

^a Sucrose initiated polyether polyol, OH number = 360.

^b 1,1,1,3,3-pentafluoropropane

20

The materials for use as the catalyst in the above formulation include gelling catalysts triethylenediamine; N-2-hydroxypropyl-triethylenediamine ammonium salt; N-cetyl-N,N-dimethylamine; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine; N-cyclohexyl-N-methylcyclohexylamine; and 1,3,5-tris(3-(dimethylamino)propyl)-hexahydro-s-triazine; and blowing catalysts bis(dimethylaminopropyl)methylamine, bis-

(dimethylamino-ethyl)ether; bis(3-dimethyl-aminopropyl)-N,N-dimethylpropanediamine and bis(N,N-dimethylaminoethyl)ether.

The ingredients in Table 1 are combined and incubated at 10°C until the mixture has equilibrated to temperature. The resin is weighed into a paper cup and the silicone
5 surfactant is added. Polymeric MDI at 23°C is added in an amount such that the isocyanate (NCO) index is 115. The mixture is stirred with a high speed mixer for 8 seconds and poured into a 12x12x2 inch (30.5x30.5x5.1 cm) mold which is heated to 49°C. Enough of the foaming mixture is added to the mold to yield a plaque which is 5% overpacked. The foam plaque is removed from the mold after 4 minutes and aged at
10 23°C for 18 to 24 hours.

CLAIMS

1. A composition comprising a polyisocyanate, a polyol, a hydrofluorocarbon blowing agent, optionally water as an additional blowing agent, a surfactant, and at least one catalyst for the reaction of the polyisocyanate with the polyol and/or the reaction of the polyisocyanate with water, the catalyst being selected from the group consisting of triethylenediamine; N-2-hydroxypropyltriethylenediamine ammonium salt; N-cetyl-N,N-dimethylamine; N,N-diethyl-ethanamine; N,N-dimethylaminoethylmorpholine; bis(3-dimethylamino-propyl)-N,N-dimethylpropanediamine; N-cyclohexyl-N-methylcyclohexylamine; 1,3,5-tris(3-(dimethylamino)propyl)hexahydro-s-triazine; bis(dimethylamino-propyl)methylamine; dibutyltin dilaurylmercaptide; dibutyltin diisooctylmaleate; dibutyltin bis(2-ethylhexylmercaptoacetate); stannous octoate, 1,2-dimethylimidazole, bis-(dimethylaminoethyl)ether; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine and bis(N,N-dimethylaminoethyl)ether and resulting in a decreased amount of decomposition of the hydrofluorocarbon blowing agent to fluoroalkene.

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2. A process for preparing polyurethane foam which comprises reacting a polyol with a polyisocyanate in the presence of a hydrofluorocarbon blowing agent, optionally water as an additional blowing agent, a surfactant, and at least one catalyst selected from the group consisting of triethylenediamine; N-2-hydroxypropyltriethylenediamine ammonium salt; N-cetyl-N,N-dimethylamine; N,N-diethyl-ethanamine; N,N-dimethylaminoethylmorpholine; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine; N-cyclohexyl-N-methylcyclohexylamine; 1,3,5-tris(3-(dimethylamino)propyl)hexahydro-s-triazine; bis(dimethylamino-propyl)methylamine; dibutyltin dilaurylmercaptide; dibutyltin diisooctylmaleate; dibutyltin bis(2-ethylhexylmercaptoacetate); stannous octoate, 1,2-dimethylimidazole, bis-(dimethylaminoethyl)ether; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine and bis(N,N-dimethylaminoethyl)ether, in

20

25


which the catalyst results in a decreased amount of decomposition of the hydrofluorocarbon blowing agent to fluoroalkene.

5

ABSTRACT OF THE DISCLOSURE

- 5 A composition comprising a polyisocyanate, a polyol, a hydrofluorocarbon blowing agent, optionally water, a surfactant, and at least one catalyst for the reaction of the polyisocyanate with the polyol and/or the reaction of the polyisocyanate with water, the catalyst being selected from the group consisting of triethylenediamine; N-2-hydroxy-propyltriethylenediamine ammonium salt; N-cetyl-N,N-dimethylamine; N,N-diethyl-
- 10 ethanamine; N,N-dimethylaminoethylmorpholine; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine; N-cyclohexyl-N-methylcyclohexylamine; 1,3,5-tris(3-(dimethyl-amino)propyl)hexahydro-s-triazine; bis(dimethylaminopropyl)methylamine; dibutyltin dilaurylmercaptide; dibutyltin diisooctylmaleate; dibutyltin bis(2-ethylhexylmercapto-
- 15 acetate); stannous octoate, 1,2-dimethylimidazole, bis-(dimethylaminoethyl)ether; bis(3-dimethylaminopropyl)-N,N-dimethylpropanediamine and bis(N,N-dimethylaminoethyl)-ether and resulting in a decreased amount of decomposition of the hydrofluorocarbon blowing agent to fluoroalkene.

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DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION (37 CFR 1.63)	Attorney Docket Number	06061 USA
	First Named Inventor	Jeffre Kramer
	COMPLETE IF KNOWN	
	Application Number	/
	Filing Date	
	Group Art Unit	
<input checked="" type="checkbox"/> Declaration Submitted with Initial Filing	OR	<input type="checkbox"/> Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)
Examiner Name		

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Catalysts Which Stabilize Hydrofluorocarbon Blowing Agents in Polyurethane Foam Compositions

the specification of which

(Title of the Invention)

☒ is attached hereto

OR

☐ was filed on (MM/DD/YYYY)

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(if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

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			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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I hereby claim the benefit under 35 U.S.C. 119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)

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[Page 1 of 2]

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U.S. Parent Application or PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)

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23543

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Name of Sole or First Inventor:

☐ A petition has been filed for this unsigned inventor

Given Name (first and middle (if any))		Family Name or Surname					
Jeffrey		Kramer					
Inventor's Signature			Date				
Residence: City	Schnecksville	State	PA	Country	USA	Citizenship	USA
Post Office Address	4012 Providence Court						
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City	Schnecksville	State	PA	ZIP	18078	Country	USA

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